

# Chromatic reintegration of historical mortars with lime-based pozzolanic consolidant products

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## Abstract

The consolidation process of old renders with loss of cohesion is nowadays performed usually with inorganic compatible products, such as calcium or barium hydroxide or ethyl silicate. The use of organic consolidants as acrylic or vinilic resins is discouraged due to its physico-chemical incompatibility with the original lime-based renders; these organic products work as adhesive in the original substrate, while inorganic consolidants contribute to recover the cohesion loss improving the binder amount and guarantying better durability and full compatibility. The restoration intervention on historic mortars, including consolidation, often causes aesthetic heterogeneity and prejudices, sometimes dramatically, the global legibility of the architectonic surface. The aesthetic function can be restored by chromatic reintegration, through several techniques, with resource to overpaints, for instance based on calcium hydroxide, pigments and silicates. In this study several consolidant lime-based products were produced and characterized. Pigments were added to the products, in order to achieve chromatic reintegration properties as well as consolidation. The consolidant products consist in aqueous dispersion of calcium hydroxide, a pozzolanic additive (metakaolin or diatomite) and mineral pigments, namely a yellow earth pigment and a synthetic red pigment. The incorporation of a pozzolanic material was made to improve the mechanical strength and durability of the binder. The consolidant products were applied on mortar specimen with a simulated cohesion loss, through a reduced binder quantity and an optimization of the aggregate grain size. Specimens were subjected to different storage conditions (50 and 95% RH), to verify the influence of curing conditions. Physico-mechanical and microstructural characterization was performed on the treated mortar specimens to verify the consolidant treatment efficacy. Consolidant treatments show interesting results, and their efficacy vary depending on the pozzolanic addition; pigments do not influence that much the consolidation efficiency.

**Keywords:** Consolidation products, historical renders, pozzolanic materials, chromatic reintegration, compatibility.

## 1. Introduction

Mortars, composed of a mix of binder and aggregates, are widely used as building materials for construction and/or decorative purposes, e.g. on historical building façade; they perform a protective function for masonry and define the aesthetic appearance of buildings.

Mortars are very versatile building product due to their plasticity, adhesion, easy application, protective and aesthetic function. However they present, mostly as external renders, a protective function and are particularly exposed to degradation agents. Mortars become friable and susceptible to material loss by several degradation phenomena. The presence of water or high humidity, salts crystallization,

action of environmental agents or occurrence of chemical phenomena can lead, among other anomalies, to adhesion and cohesion loss (Toniolo 2010, Borsoi 2012a).

The recovery of the cohesion is achieved with the application of consolidant products that penetrate on the porous structure of the mortar and restore the connections of the binder-aggregate system. Inorganic products, such as limewater, barium hydroxide or ethyl silicates, show compatibility with old renders and higher durability than organic consolidants.

In order to achieve a durable treatment, important consolidant products requirements have to be considered, such as a mechanical improvement and the respect for the water absorption properties of the treated mortars; furthermore chromatic alterations of the original render must be avoided (Toniolo 2010, Hansen 2003), since the reproduction of the original chromatic aspect in treated surfaces is a common problem for restorers (Candelaria 2008a). The repair of renders, by fulfilling lacunae and cracks, generally causes aesthetic heterogeneity and compromises the global legibility of the architectonic surface, sometimes dramatically.

This study focuses on the development and characterization of consolidant lime-based products with pozzolanic additives. The possibility of adding chromatic reintegration functions to those products by addition of mineral pigments is also studied.

Two lime-based consolidants, with the addition of metakaolin (MK) or diatomite (DM), were studied; MK and DM are well-known materials that react with  $\text{Ca(OH)}_2$ , at room temperature in humid conditions, to form compounds with hydraulic properties (ASTM 2003). Furthermore the consolidant products were modified with the addition of two inorganic pigments, a yellow natural earth and a red synthetic iron oxide, in order to evaluate the viability of simultaneous chromatic reintegration. The experimental procedure allows the evaluation of the viability and efficacy of these intervention processes, and more specifically the simultaneous consolidation and chromatic reintegration. The reintegration must not significantly change the functional characteristics of the surface and must be aesthetically adequate, both in new state and after ageing.

## 2. Materials

### 2.1 Mortar specimens

Several mortar specimens were prepared varying the binder/aggregate ratio (in volume) from 1:8 to 1:3 (Tavares 2008), in order to formulate a mortar with simulated cohesion loss. The binder used was a commercial hydrated lime CL 90 available in Portugal. A well graduated siliceous sand was used, simulating those aggregates commonly adopted for render mortars. The aggregates were composed of a mixture of 3 different calibrated sands (mean particle sizes < 2mm). After preliminary experimentation, a binder/aggregate ratio of 1:4 (in volume) was selected as it guarantees the desired effect of a low cohesion mortar without significant loss of material. The water:binder ratio selected was 2:1 (mass) (Borsoi 2012a). Different samples were prepared with the selected mortar, namely prismatic specimens (40x40x160 mm) and ceramic bricks (28x19 cm) with a single mortar layer of 1.5 cm thickness.

### 2.2 Consolidant products

#### 2.2.1 Materials and composition

The products were produced mixing calcium hydroxide, metakaolin (MK) or diatomite (DM), distilled water and two mineral pigments in two different concentrations (Table 1). The lime was the same used for the mortar specimen production.

MK and DM are both artificial pozzolanic materials, thermally activated, with notable potentialities due to their high amorphous aluminosilicate (MK) or silica (DM) content. In this study two commercial pozzolanic materials were used, the metakaolin Argical M-1200S and the diatomite Celite 209, both commercialized by Imerys. DM presents a content of 82% silica and 7.3% of CaO and an average grain size of 10  $\mu\text{m}$ , while MK has a high amount of silica (55%) and alumina (39%) and average grain size of 1-2  $\mu\text{m}$ . Modified Chapelle test was performed to determine the pozzolanic activity according to the French NF P18-513 standard. This test concerns the measure of the calcium hydroxide fixed by metakaolin when in contact with a saturated solution of calcium hydroxide for a period of 16 hours at a temperature of  $85 \pm 5^\circ\text{C}$  (AFNOR 2010, Pontes 2013). The pozzolanic activity of the MK and DM, expressed in mg of  $\text{Ca(OH)}_2$ , was respectively 1293 mg  $\text{Ca(OH)}_2/\text{g}$  and 1349 mg  $\text{Ca(OH)}_2/\text{g}$ .

Two mineral pigments were selected for chromatic reintegration, a yellow natural yellow ochre, identified as *Alvalade* (YA), and a red synthetic ochre, identified as *Bayer* (RB). Their colour is correlated with the presence of iron oxides, mainly goethite ( $\text{FeO}(\text{OH})$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ), that respectively represent the main chromophore for yellow and red ochres. Goethite is considered the most abundant iron oxide in nature (Eastaugh 2004, Elias 2006). XRD analysis confirms that yellow *Alvalade* and red *Bayer* are pure iron oxides, containing respectively goethite and hematite (Candelaria 2008b). The pigments were chosen considering their mineralogical composition and the colors reproduction on mortars specimens.

Table 1 Consolidant products identification and composition, where MK = Metakaolin; DM= Diatomite;  
YA = Yellow *Alvalade*; RB = Red *Bayer*.

Identification	Products composition	Dry residue (g/L)	pH
MK	Lime + MK (25%) + $\text{H}_2\text{O}$	Vary from 75 to 80 g/L	12.1 – 12.5
MY5	Lime + MK (25%) + 5% YA + $\text{H}_2\text{O}$		
MR5	Lime + MK (25%) + 5% RB + $\text{H}_2\text{O}$		
DM	Lime + DM (25%) + $\text{H}_2\text{O}$		
DY5	Lime + DM (25%) + 5% YA + $\text{H}_2\text{O}$		
DR5	Lime + DM (25%) + 5% RB + $\text{H}_2\text{O}$		

Based on previous experimentation (Borsoi 2012b), lime binder was replaced in 25% (weight %) by MK or DM in order to induce the formation of pozzolanic products and to minimize the filler effect. Mineral pigments were added in a reduced concentration (5%, calculated on the weight of the dry solid materials), commonly adopted for chromatic reintegration of historical render. A 1:4 solid:liquid ratio was selected since it guarantees an efficient applicability and a suitable dry residue (Borsoi 2012b).

### 2.2.2 Preparation and application

Consolidant products were prepared by magnetic stirring of the different components until complete homogenization. Consolidants were nebulized in ten consecutive applications both on prismatic specimens and on brick rendered surfaces, in order to achieve homogeneous distribution and moderate surface consolidation; they were applied from a distance of 20-30 cm in a controlled environment.

After consolidants application, specimens were stored for 7 days in a climatic chamber with humid conditions at  $T=20\pm 2^\circ\text{C}$  and  $95\pm 5\%$  RH in order to promote the formation of pozzolanic products (Gameiro 2012). Treated specimens were then stored in a conditioned room with  $20\pm 2^\circ\text{C}$  and  $65\pm 5\%$  RH (close to real external conditions), to allow the carbonation of the free lime still existent.

## 3. Test methods

A series of analytical techniques were performed on treated and untreated specimens in order to assess the consolidant products efficacy and to characterize the consolidation treatments. The analytical procedures were performed at 28 and 90 days after consolidants application, and then after accelerated aging test.

Flexural ( $R_f$ ) and compressive ( $R_c$ ) strength tests were performed with an ETI-HMS apparatus, according to the EN 1015:11 (CEN 1999). The superficial hardness of mortars was determined with a durometer Shore A, based on the ASTM D2240 (ASTM 2000). This equipment measures the penetration resistance of a metallic pin on the analyzed material. Water absorption properties were verified with Karsten tubes using the applications of mortar on bricks specimens. The procedure performed is based on a methodology adopted by RILEM (RILEM 1980). Freshly fractured surfaces of mortar specimens and the consolidants penetration depth were observed with an Olympus SZH microscope; images were recorded with Olympus DP-20 and SC-30 digital microscopy cameras. Consolidants penetration depth

was verified by observing the cross section of the treated mortars, measuring the average mean of at least 12 point for each treatment, using an interactive measurement tool of the Olympus LabSens software.

Finally an accelerated aging test was performed on treated mortar specimens to verify the treatments resistance to aggressive environmental conditions. Mortars applied on ceramic bricks were subjected to heat-cold and freeze-thaw cycles, and test conditions are presented in Table 2. Temperature cycles (heat-cold) were performed using a set of infrared heat lamps (250W power/each lamp) and a deep freeze cabinet set. Simulation of rain and subsequent freeze / thaw cycles was achieved by exposing the specimens to a sprinkler system. Afterwards the specimens were stabilized and inserted in the deep freeze cabinet. The chosen conditions were adapted from the EN 1015:21 (CEN 2002) and represent extreme climate conditions. Eight temperature and freeze-thaw cycles were performed instead of the four as indicated in the European Standard.

Table 2 Accelerated aging test conditions.

Temperature cycles	Freeze-thaw cycles	Exposure time (h)
Infrared lamps ( $60\pm5^{\circ}\text{C}$ )	Sprinkler system (simulated rain)	8
Stabilization ( $20^{\circ}\text{C}$ , 65%RH)		$\frac{1}{2}$
Deep freeze cabinet ( $-8^{\circ}\text{C}$ )	Deep freeze cabinet ( $-8^{\circ}\text{C}$ )	15
Stabilization ( $20^{\circ}\text{C}$ , 65%RH)		$\frac{1}{2}$

## 4. Results and Discussion

### 4.1 Flexural and compressive strength

Figure 1 shows the mechanical test results of untreated and treated specimens, analyzed at 28 and 90 days after consolidation. Three prismatic specimens of each treatment were analyzed for flexural strength ( $R_f$ ); the tensile strength was practised on the consolidated surface. The compressive strength ( $R_c$ ) analysis was performed on each 'half-specimen' obtained from the flexural strength analysis, being the value reported an average of 6 specimens/treatment.

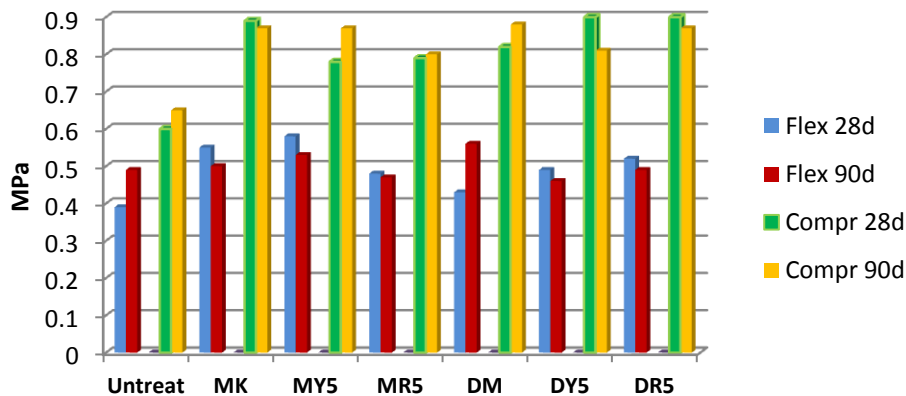


Figure 1 Flexural ( $R_f$ ) and compressive ( $R_c$ ) strength results.

MK-based treatments illustrate mechanical improvement at 28 days, but these values decrease at 90 days; pigment additions illustrate a slight influence, with values similar to MK treatment, causing either mechanical reduction or improvement, depending on the pigment used. The maximum reduction verified is about 15% (MR5) for flexural strength, and MR5 consolidants reached lower values in both flexural and compressive strength, compared to MK treatment. MY5 treatment however shows higher mechanical values compared to MK treatment, both on flexural and compressive strength. These can be linked to goethite reactivity, related to its surface functional hydroxyl groups, with air lime in an aqueous dispersion (Marshall 2005).

DM-based treatments illustrate a higher influence of pigment addition, since a notable flexural and compressive strength decrease is evident at 90 days analysis. On the other hand DM treatment confers good values at 90days.

Consolidation treatment should moderately improve the flexural and compressive strength, in general not exceeding 1.5 times the values of untreated specimens (Toniolo 2010); MK-based and DM-based treatments show an improvement of mechanical characteristics; the pigmented consolidants evidence small mechanical increase, and sometimes a slight loss of flexural strength is observed, as in the case of MR5, DY5 and DR5.

#### 4.2 Durometer hardness (Shore A)

Measurements were performed on mortars applied on ceramic bricks, with 12 measurements/specimen. Figure 2 shows the superficial hardness results of untreated and treated mortars, analyzed at 28 and 90 days after consolidation treatment, as well as the results after accelerated aging test.

Superficial hardness values illustrate an improvement of treated specimens compared to mortar reference. The results of treated specimens are all quite close, with a moderate improvement of superficial hardness (average 10%) comparing to the untreated mortar. A slight increase of superficial hardness from 28 to 90 days is noticeable with MK-based consolidants while DM-based consolidants illustrate very variably slight increase or decrease; after accelerating aging the untreated specimen suffered a significant loss of superficial hardness; on the other hand MK-based consolidants avoided loss of strength and DM-based products permitted a moderate decrease (8 to 19%), therefore indicating a lower durability to climatic cycles than MK-based products. The addition of inorganic pigments did not yield a significantly negative influence on the superficial hardness, especially for MK-based treatments, whereof results remained nearly identical after accelerated aging test. However DM-based consolidants evidence lower durability when yellow or red pigments are included, respectively of 19% and 11% compared to hardness values at 90 days.

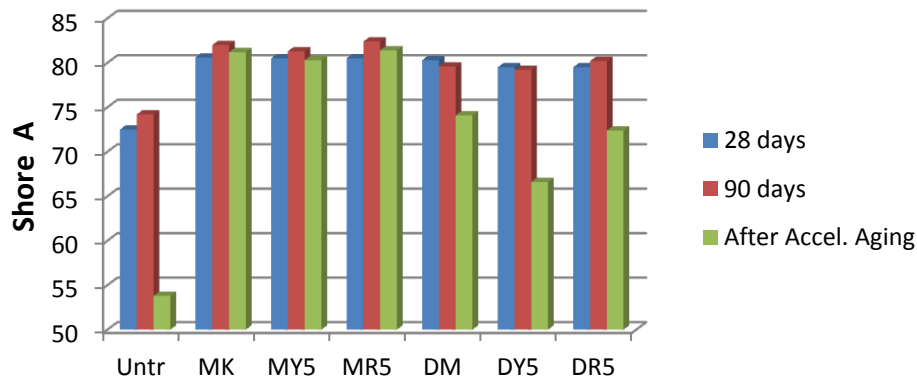


Figure 2 Superficial hardness results (average values).

#### 4.3 Karsten tubes

Karsten pipes were applied on untreated and treated specimens, to verify the water absorption variation due to consolidation treatments; this test was performed on the mortars applied on bricks and simulates the pressure of above 140 km/h wind on the vertical surface of the specimens where pipes are applied (RILEM 1980). Two Karsten pipes were glued with mastic adhesive to each specimen, testing 3 specimens/treatment. Time values (in seconds) for total absorption of 4 ml of water were registered at 28 and 90 days and after accelerating aging (figure 3). The reduction of water permeability is evident in all treated specimens; absorption times of MK-based treatments were more than 10 times higher than untreated specimens, reaching 15 to 20 times for DM-based consolidants.

DM-treatments achieve higher variation of the water absorption properties (capillary suction), probably due to diatomite filler effect; anyway their lower durability is evident since 90 days values decrease dramatically after accelerating aging. Values underline a notable permeability decrease already at 90 days, compared to 28 days results.

MK-based treatments instead evidence heterogeneous values but higher durability at heat-cold and freeze-thaw cycles, with small decrease of water absorption properties after aging. Pigmented consolidants reflect almost the same values; yellow and red pigments do not affect that much water absorption, with values similar to their respective unpigmented products; once more MK-treatments evidence higher durability also with pigment addition. For both pozzolanic materials, yellow earth addition confers slight higher permeable treatments, compared to red pigment addition.

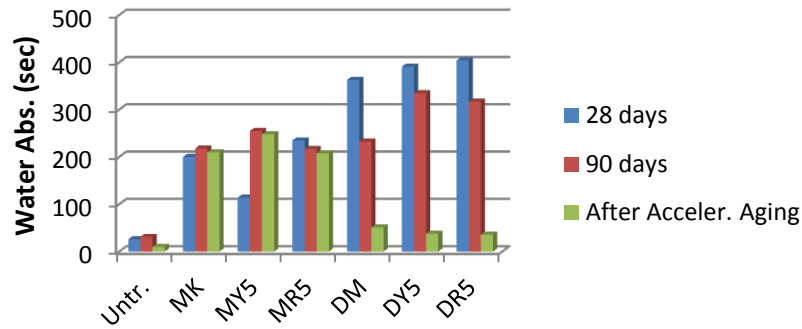


Figure 3 Karsten tubes test results (average values).

Results evidence high variability and standard deviations due to the high heterogeneity and porosity of the treated mortars. However, the alteration of water transport properties (capillary suction) is moderate and this slight hydrophobic behaviour could be beneficial if it does not change drastically the vapour permeability of the original render/mortar.

#### 4.4 Stereozoom microscopy observations

##### 4.4.1 Freshly fractured surfaces

Treated mortars present a compact surface and an evident pore decrease and infilling, compared to untreated mortar (Figure 4). These alterations are linked to the possible presence of hydraulic neoformation products on the treated surface, and to the filler effect of MK and DM.

Concerning MK-based products (Fig. 4b), consolidants do not create a superficial layer on the treated mortar, avoiding drastical changes in the water absorption properties as seen in section 4.3, due to the reduced grain size average (1-2  $\mu\text{m}$ ) of metakaolin.

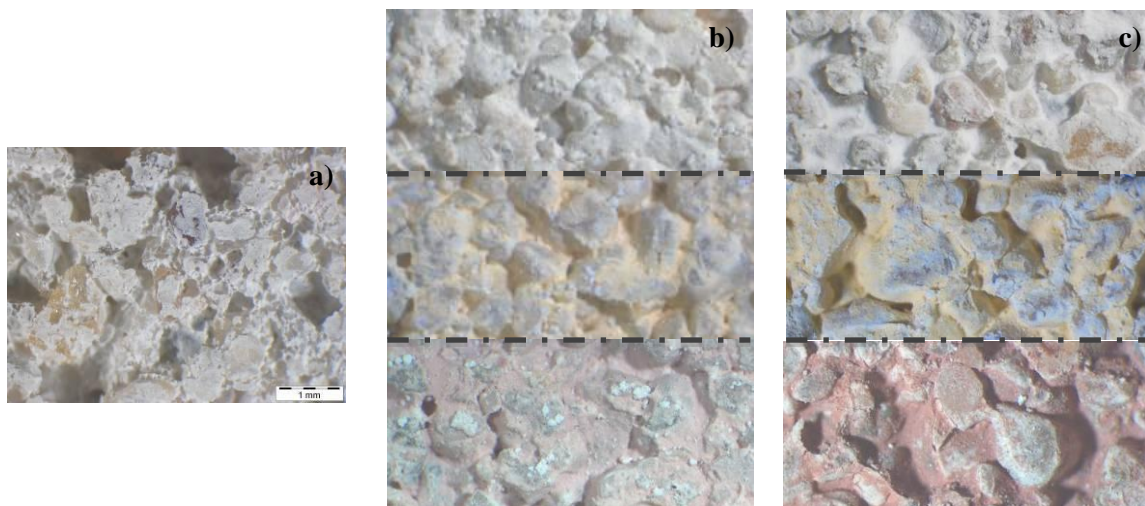


Figure 4 Stereozoom microphotographs (20x) of (a) untreated mortar specimens, (b) specimens treated with MK, MY5 and MR5, and (c) specimens treated with DM, DY5 and DR5.



DM-based treatments (Fig. 4c) evidence a more compact surface, with a more uniform distribution compared to MK treatments; the infilling of the superficial mortar pores induce, as seen in section 4.3, an alteration of mortar capillarity suction, and these microscopical observation could justify that issue. DM treatment bestows higher whitening compared to MK treatment. Besides the higher particle size (10  $\mu\text{m}$ ), compared to MK, DM is known for its optical properties and high light scattering (Barbero Barrera 2011) justifying this white glittering patina on the surface.

#### 4.4.2 Penetration depth

The penetration depths of the consolidants were analyzed observing the freshly fractured cross section of the treated mortar, measuring a minimum of 12 points/sample. Penetration depth values are reported in table 3. Figure 6 illustrates the penetration depth of MY5 and MR5 treatments.

MK treatments present heterogeneous depth distribution, due to mortar heterogeneity, varying in a large range between 1.2 mm and 5.5 mm. DM treatments highlight as well heterogeneous distribution (1.0 to 4.7 mm) but the penetration depth is slightly lower compared to MK treatments. This value was predictable, due to higher DM particle size and to the previous optical observations that underline a superficial patina on the treated surface (section 4.4.1).

Yellow and red pigments behave in a similar manner, presenting minimal differences in the consolidant penetration depth. These values are referred just to pigmented treatments and, compared to MK or DM, the pigment addition may affect the penetration due to the product composition and dry residue increase.

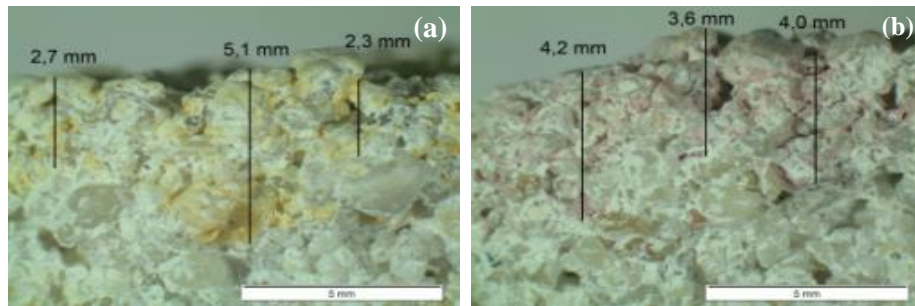


Figure 6 Microphotographs showing penetration depth measurements of (a) MY5 and (b) MR5 treatments.

## 5. Conclusions

Consolidant products with pozzolanic characteristics considered in this work confer a moderate mechanical improvement to the treated lime mortars. MK-based consolidants show some differences compared to DM-based products.

MK-consolidants illustrate a low mechanical decrease at 90 days analysis; as seen in previous work (Gameiro 2012) some pozzolanic compounds are known to be metastable. DM-consolidants instead indicate higher values at 90 days compared to 28 days analysis. Nevertheless, accelerating aging test show that durability of MK-treatments is higher, since mechanical values for DM-treatments drastically decrease after artificial aging. Values at 180 and 360 days should be considered in future work to assess the evolution over time of these treatments. Pigments seem to interfere slightly both on compressive/flexural strength and superficial hardness, reducing these values. Water absorption results evidence that DM-treatments induce significant alteration of water capillarity suction, compared to MK-treatment, but also in this case, after artificial aging, water absorption values decrease, evidencing a low durability of DM-treatments. Pigments do not have a notable influence on water absorption properties, conferring a slight increase of capillarity suction probably due to pigments filler effect.

Inorganic pigments addition did not show a consistent decreasing influence on the consolidant products efficacy. Compared to the ochre earth pigment (goethite), the synthetic red iron oxide (hematite) presents higher chromatic stability. Specimens treated with MK-based consolidant appear well-resistant to temperature cycles, showing just minimal chromatic variations in pigmented treatments (whitening).

DM-based products illustrate a reduced durability after accelerating aging, as evidenced in the drastic decrease of superficial hardness values (section 4.2). After freeze-thaw cycles, treated specimens showed superficial cracking with reduced material loss. Untreated specimens showed instead severe degradation and material loss, since just around 25% of the initial mortar specimen was preserved after 8 cycles.

Microscopically all treatments confer homogenous consolidation with partial pore infilling; DM-treatments produce slight whitening to the treated surface, and more glittering colours concerning pigmented treatments, compared to MK-treatments. Concerning chromatic reintegration, pigmented consolidant products conferred homogeneous appearance on mortar surface and demonstrate to be stable after accelerated aging, appearing well-fixed in the consolidant treatment. Penetration depth of the consolidant products is heterogeneous, varying from 1 to 5 mm for both treatments, nevertheless MK-treatments demonstrate slightly higher penetration depth due probably to its thinner grain size, compared to DM-treatments. The possibility to act simultaneously on chromatic reintegration and consolidation intervention represents significant potential benefits for the conservation of coloured historical renders, that said, it must be emphasized that additional work is still required in this area.

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